

PATENT APPLICATION Mo-6323 STA-151

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION OF	) ) GROUP NO.: 1742
GERHARD GILLE ET AL	) )
SERIAL NUMBER: 09/831,567	) EXAMINER: H.D. WILKINS III
FILED: MAY 10, 2001	) ) `
TITLE: METHOD FOR PRODUCING WOLFRAM CARBIDES BY GAS-PHASE CARBURETION	, ) ) )

## **DECLARATION UNDER 37 C.F.R. §1.132**

- I, Dr. Benno Gries, residing at Wolfenbuettel, Germany, declare as follows:
- 1) that I have the following technical educations and experience:
  - a) I am a chemist having studied at Hannover Germany, from 1978 to 1985,
  - b) I received the degree of \_Ph.D. at the Hannover University in the year of 1988.
  - c) I am employed by Bayer, H.C. Starck GmbH, since 11/1988 in the R&D department, in particular with regard to development of refractory metal and carbide powders.
- 2) that the following tests were carried out under my immediate supervision and control:

The Examples were carried out as described in Example 1 of the subject patent application. The CO/CO2 ratio and the carbon activity resulting there from are summarized in the following table:

Experiment	Ratio CO/CO <sub>2</sub>	Carbon activity	Total C content (%)	Free C content (%)	O (%)	Product comp. following materials
Comparative Example 1	93.9 : 6.1	0.35	3.62-3.83	< 0.02	0.0877	W, WC, W <sub>2</sub> C
Comparative Example 2	98.2 : 1.8	1.11	5.87-5.93	< 0.02	0.1894	W, WC
Comparative Example 3	99.0 : 1.0	1.03	5.96	< 0.02	0.2223	W, WC

The materials comprised in the product were characterized by X-ray diffraction. As can be seen in Comparative Example 1, the reaction was not complete yet and contained only 63% of tungsten carbide. Comparative Examples 2 and 3 show better conversion rates, but still comprise some intermediate tungsten. Total and free carbon level are comparable to Example 1 of the application, but, additionally, loose fluffs of carbon black (free carbon) developed under these reaction conditions. Carbon black deposited near and outside the boat carrying the product. No carbon black developed yet inside the product, so product analysis shows only a low content of free carbon. Such carbon fluffs did neither develop under the exact conditions of Example 1 nor Comparative Example 1.

It can be seen that the excellent results in a process according to the invention as already shown in Example 1 can not be achieved when different reaction conditions are chosen.

I further declare that all statements made herein are of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States code and that such willful false statements may jeopardize the validity of pending Application Serial Number 09/831,567 or any patent issuing thereon.

Signed at Goslar, this 6<sup>th</sup> day of September, 2005.

Benno Gries